

DAMAGE ACCUMULATION IN SiC/SiC COMPOSITES WITH 3D ARCHITECTURES

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The formation and propagation of multiple matrix cracks in relatively dense ceramic matrix composites when subjected to increasing tensile stress is necessary for high strength and tough composites. However, the occurrence of matrix cracks at low stresses may limit the usefulness of some non-oxide composite systems when subjected to oxidizing environments for long times at stresses sufficient to cause matrix cracking. For SiC fiber-reinforced composites with two-dimensional woven architectures and chemically vapor infiltrated (CVI) SiC matrix and melt-infiltrated (MI) Si/SiC matrix composites, the matrix cracking behavior has been fairly well characterized for different fiber-types and woven architectures. It was found that the occurrence, degree, and growth of matrix cracks depends on the material properties of the composite constituents as well as other physical properties of the composite or architecture, e.g., matrix porosity and size of the fiber bundle. In this study, matrix cracking in SiC fiber reinforced, melt-infiltrated SiC composites with a 3D orthogonal architecture was determined for specimens tested in tension at room temperature. Acoustic emission (AE) was used to monitor the matrix cracking activity, which was later confirmed by microscopic examination of specimens that had failed. The determination of the exact location of AE demonstrated that initial cracking occurred in the matrix rich regions when a large z-direction fiber bundle was used. For specimens with large z-direction fiber tows, the earliest matrix cracking could occur at half the stress for standard 2D woven composites with similar constituents. Damage accumulation in 3D architecture composites will be compared to damage accumulation in 2D architecture composites and discussed with respect to modeling composite stress-strain behavior and use of these composites at elevated temperatures.

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Introduction

- Why 3D orthogonal composites?
 - Improved interlaminar properties?
 - Ojard et al., Ceram. Eng. Sci. Proc. (2002)
 - Improved strength due to straight fibers?
 - Improved through-thickness thermal conductivity?
- 3D orthogonal composites with different Z fibers (Yun and DiCarlo, 2002):
 - Sylramic or Sylramic-IBN fibers
 - BN interphase
 - CVI SiC
 - MI SiC (slurry-infiltrated SiC + melt-infiltrated Si)

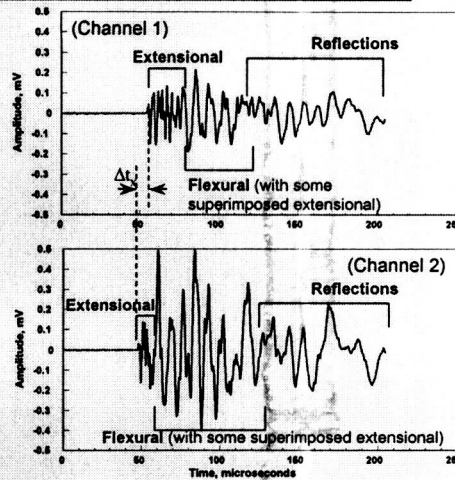
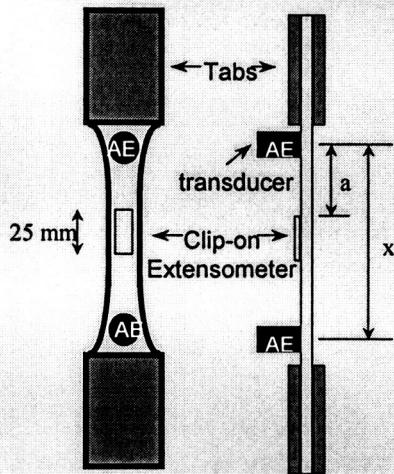
Focus of this study: Matrix crack formation

- Variety of different local architectures in series:
 - Cross-ply (XPLY) sections
 - Unidirectional (UNI) sections
- Different tow dimensions
- Different type, size, and orientation of Z fiber bundles
- Large fiber-rich and matrix-rich regions

Outline

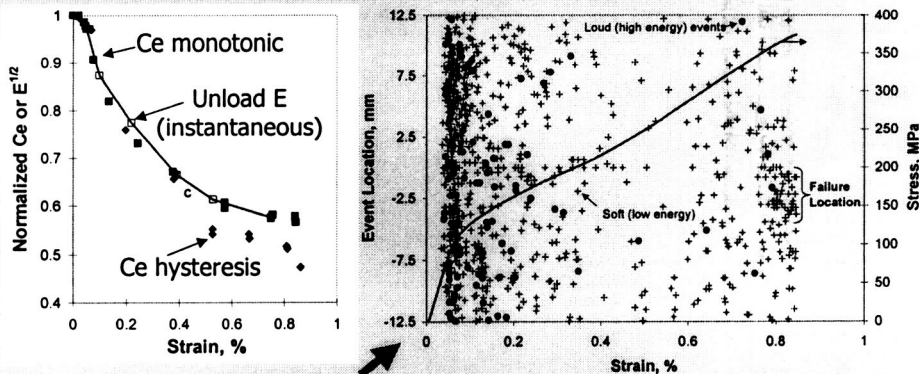
- Use of AE to monitor matrix cracks in 2D, 5 harness satin composites
- Description and tensile results of 3D-orthogonal composites tested
- Stress-induced matrix cracking in the 3D-orthogonal composites
- Comparison of matrix cracking between 3D and 2D composites

Modal Acoustic Emission of CMCs



- Locate damage events and failure events → Δt
- Monitor stress(or time)-dependent matrix cracking → Cumulative AE Energy
- Identify damage sources, e.g. matrix cracks, fiber breaks → Frequency
- Measure stress(or time) dependent Elastic Modulus → Speed of sound

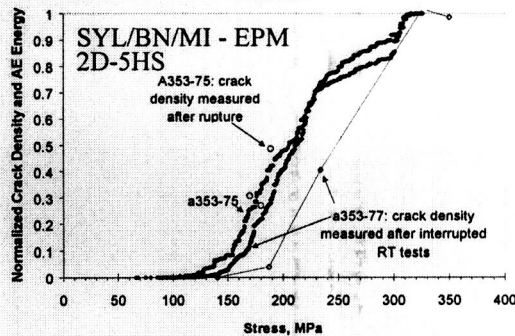
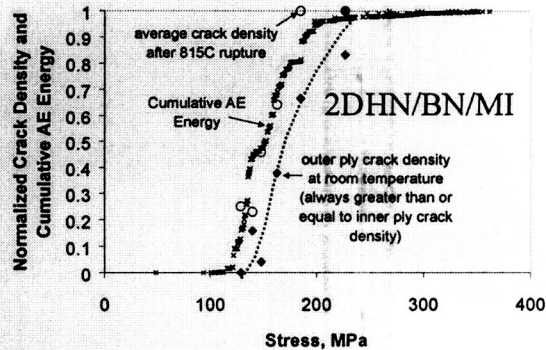
Use of AE to Monitor Matrix Cracking in CMC's



Normally, using a threshold voltage technique for location gives $\sim \pm 2$ mm accuracy. For this study, each event was examined "by hand" to determine 1st peak (± 0.5 mm)!

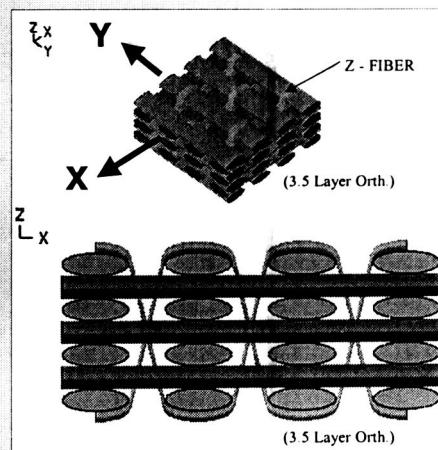
Nearly Direct Relationship Between Crack Density and AE Energy

Crack densities measured after RT interrupted tests (plasma etched polished sections) or after 815°C stress-rupture (environment opens cracks) correlate well with AE energy.



3D Orthogonal Composites

- 0° and 90° fibers were Sylramic-iBN or Sylramic
 - $\perp$ to Z fiber alignment: Y direction
 - 18 or 20 epi – **8 Plies**
 - $\parallel$ to Z fiber alignment: X direction
 - 10 double tow ends per inch, i.e., 20 epi [only tested for Double ZMI] – **7 Plies**
- Z fibers: Double ZMI; Single ZMI; T-300; Rayon



Properties

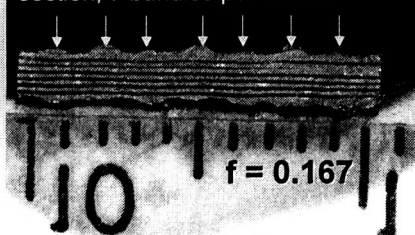
Z-Fiber Tow	Ends Per Inch	E, GPa	Failure σ , MPa	Failure Location	Debonding*
Double ZMI (Y-direction)	18	248	317	Gage	Inside
Double ZMI (X-direction)	10(2) = 20	205	322	Radius	Inside
Single ZMI (Y-direction)	20	190	311	Gage	Outside (~1mm pull-out)
T300 (Y-direction)	20	237	345		Outside
Rayon (Y-direction)	20	238	336	Radius	Outside

* **Inside** = between the BN and the fiber surface; **Outside** = between the BN and the CVI SiC matrix

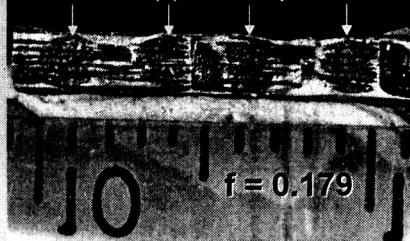
- All composites contain Sylramic-IBN fibers except for Z = Rayon composites which are reinforced with as-produced Sylramic fibers

Cross-Sections

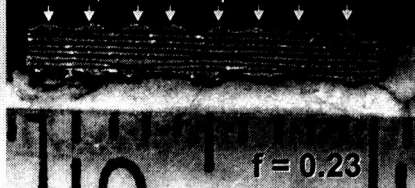
Double ZMI – Y-direction cross-section, 7 bundles per 10 mm



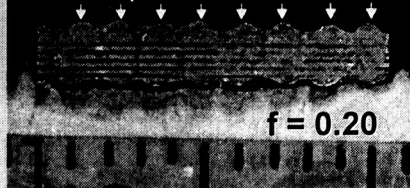
Double ZMI – X direction fract. Surface, 4x2 (8) bundles per 10 mm



Single ZMI – Y-direction cross-section, 8 bundles per 10 mm

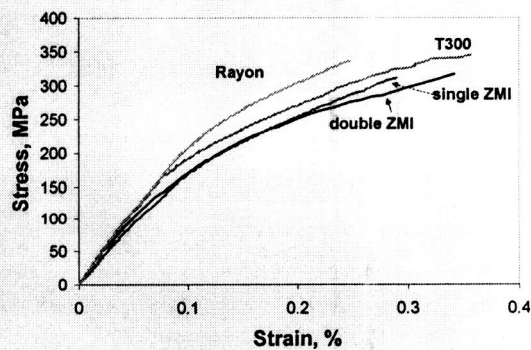
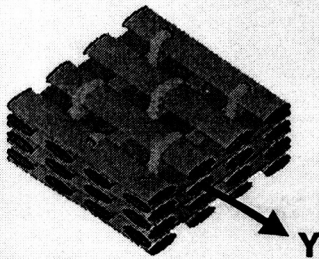
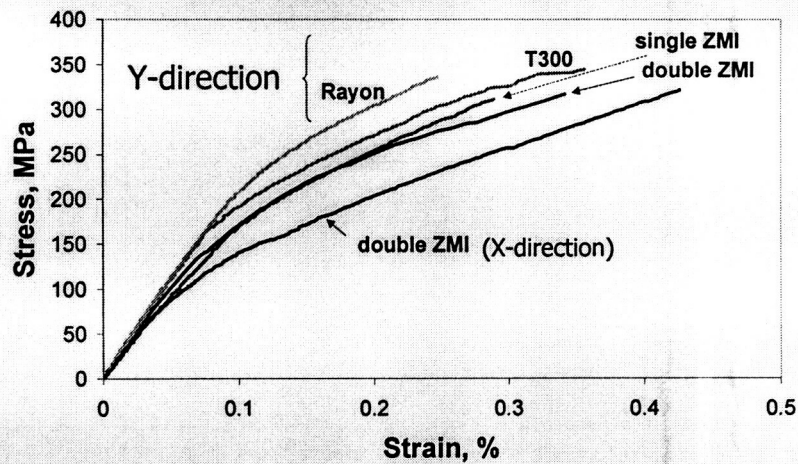


Rayon – Y-direction cross-section, 8 bundles per 10 mm



"f" is determined from area of fibers in loading direction based on epi and the thickness measurement used for the calculation of stress.

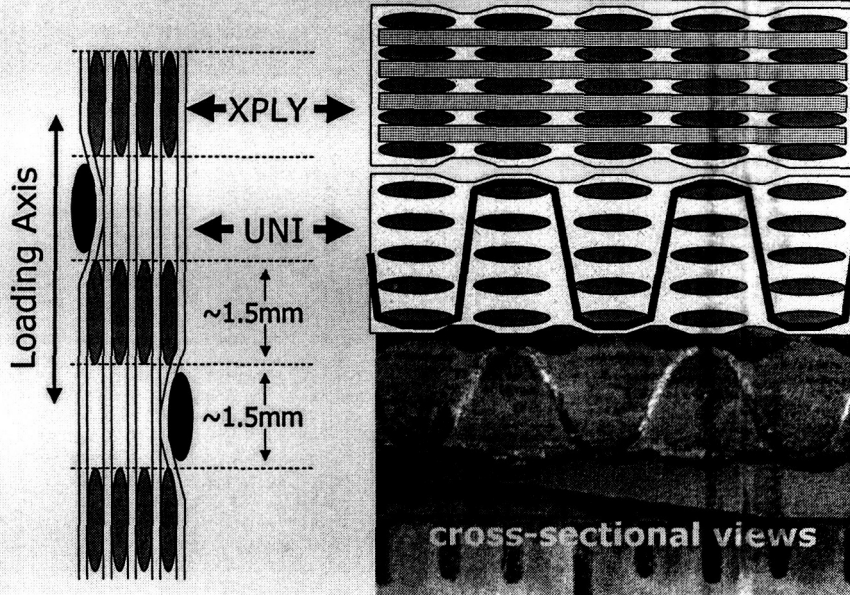
Comparison of Stress-Strain Behavior



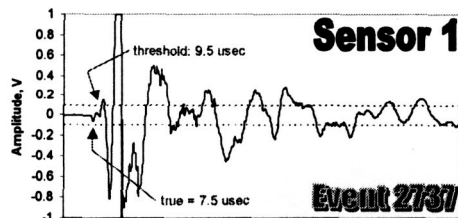
Loading in the Y Direction

Perpendicular to the Z-bundle orientation

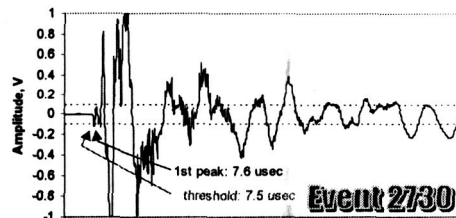
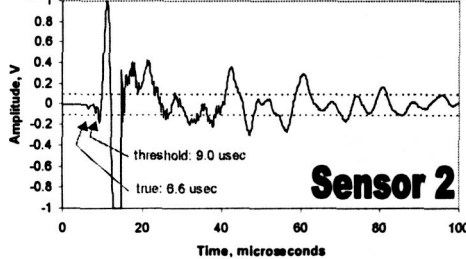
Idealized 3D Orthogonal Composite (Y-direction)



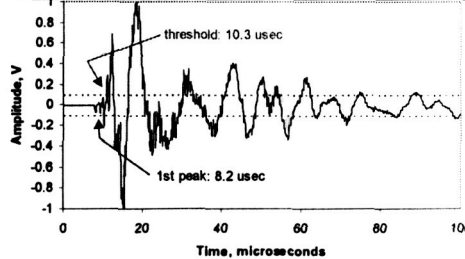
"Labor Intensive" AE Event Location Technique



Location (threshold) = + 2.2 mm
Location (peak) = + 3.4 mm

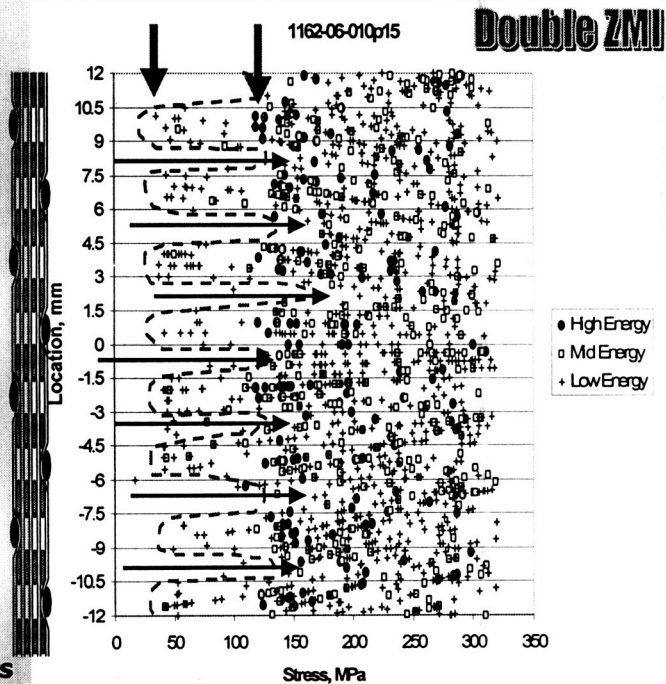


Location (threshold) = + 12.3 mm
Location (peak) = - 2.6 mm



Matrix Cracking in the Gage Section

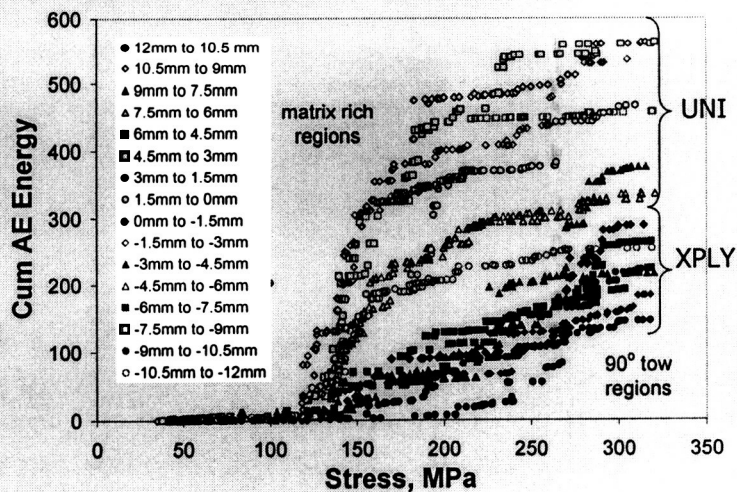
- (1) Matrix micro-cracks originate in the UNI sections (low energy AE)
- (2) Large matrix cracks form in the UNI sections (High energy AE)
- (3) Matrix cracks form in XPLY regions



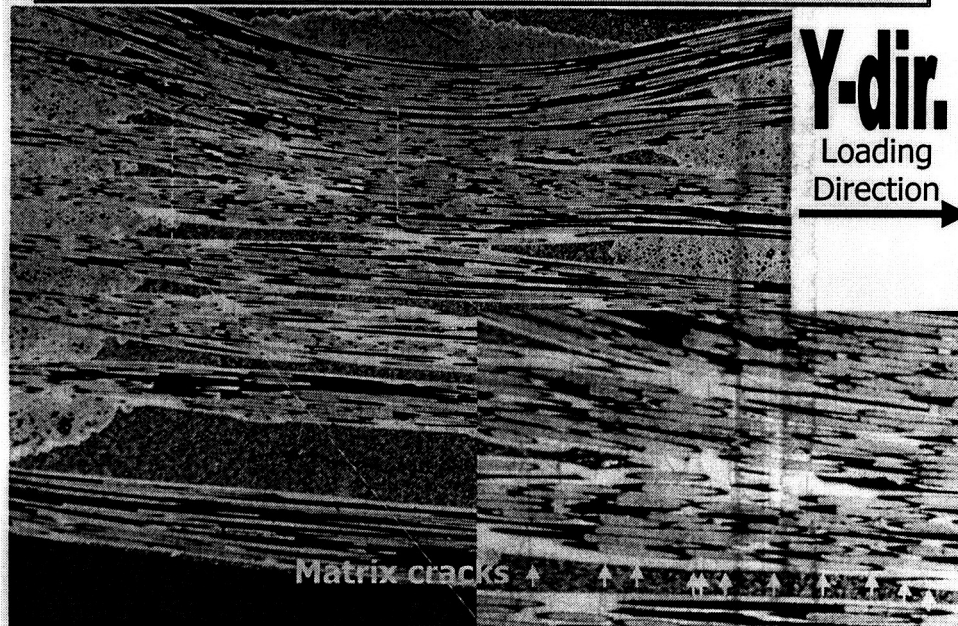
Stress-Dependent Matrix Crack Formation Differs for UNI and XPLY Regions

Cracking occurs over a narrower stress range in "UNI" region compared to "XPLY" region.

AE energy is higher for UNI regions than for XPLY: Higher fracture energy matrix



3D Orthogonal: Z = Double ZMI



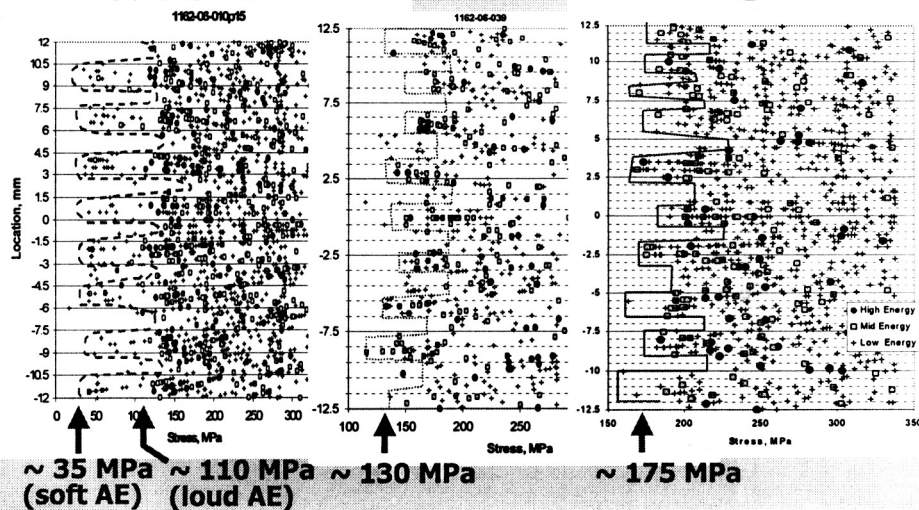
Onset of Matrix Cracking Depends on size of Z-Bundle

... ➔ Decreasing Z-bundle Size ... ➔

Double ZMI

T-300

Rayon

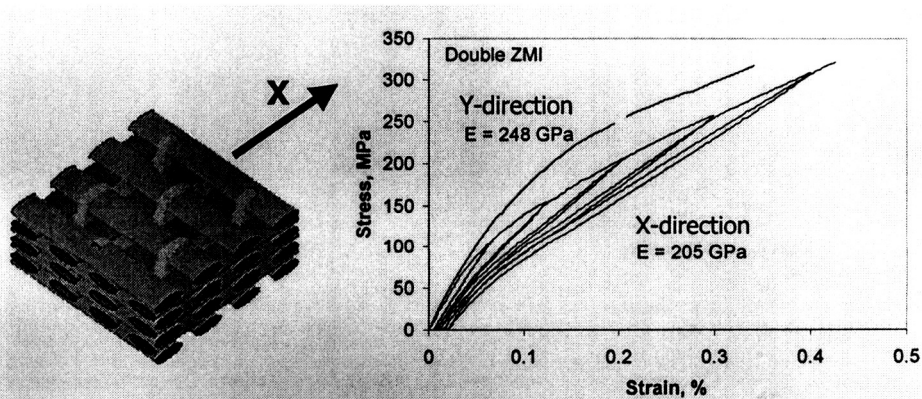


3D Orthogonal: Z = Rayon

Polished and plasma-etched



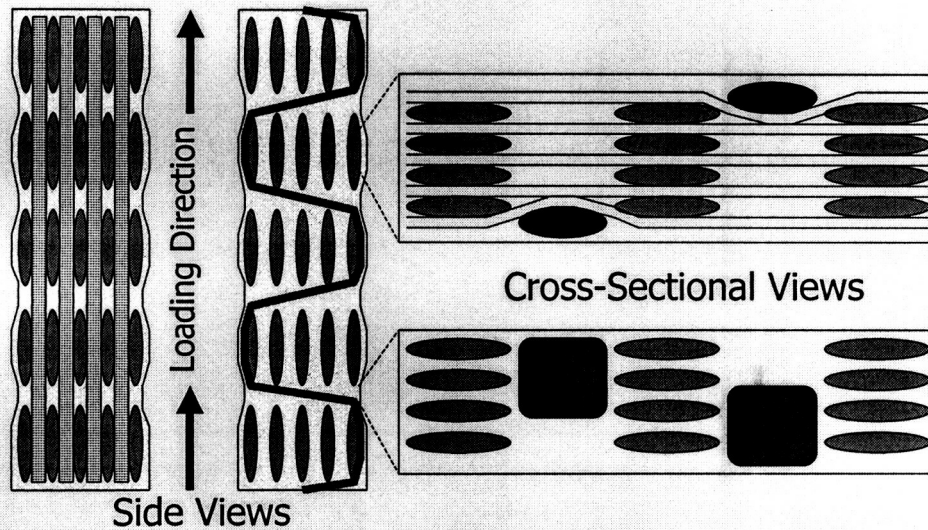
Y-direction



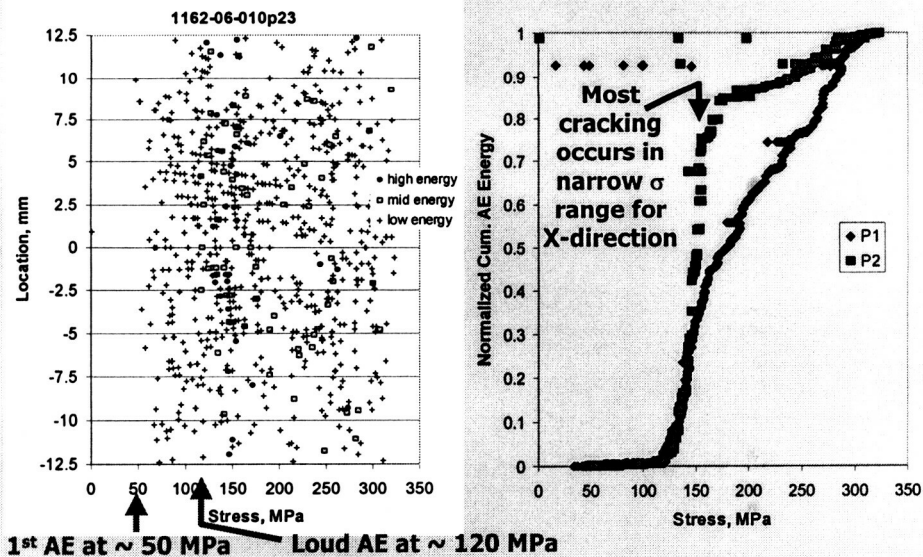
Loading in the X-Direction

- Parallel to the Z-bundle orientation
- 10 Double Sylramic-iBN tows per inch in loading direction

Idealized 3D Orthogonal Composite (X-direction)



Acoustic Emission for X- direction Stress-Strain



3D Orthogonal: Z = Double ZMI



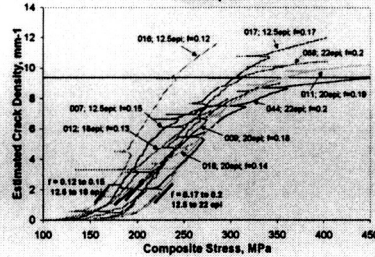
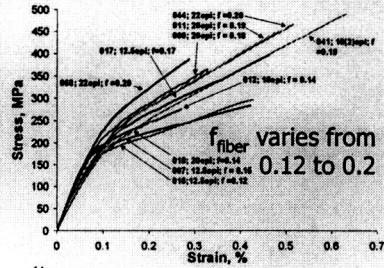
Measured Crack Densities

Specimen	UNI ρ_c mm ⁻¹	XPLY ρ_c mm ⁻¹	Avg ρ_c mm ⁻¹
Double ZMI (Y-direction)	10.2	8.8	9.5
Double ZMI (X-direction)	12	7.2	7.9
Single ZMI (Y-direction)	4.5	3.6	4.1
T300 (Y-direction)	7.4	4.3	5.9
Rayon (Y-direction)	4.8	4	4.4

ID

OD

Matrix Cracking in 5HS 2D Architectures (Based on AE and measured Crack Density)

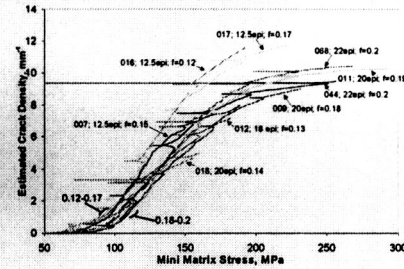


$$\sigma_{\text{minimatrix}} = \frac{(\sigma_c + \sigma_{th})}{E_c} \left(\frac{E_c - f_{\text{mini}} E_{\text{mini}}}{1 - f_{\text{mini}}} \right)$$

Assumption: Cracks originate outside of 0° minicomposite

f_{mini} and E_{mini} determined from fiber weight and weight gain of BN and CVI SIC and respective moduli and densities

E_c determined from s/e curve



Composite Properties

$$\sigma_{\text{minimatrix}} = \frac{(\sigma_c + \sigma_{th})}{E_c} \left(\frac{E_c - f_{\text{mini}} E_{\text{mini}}}{1 - f_{\text{mini}}} \right)$$

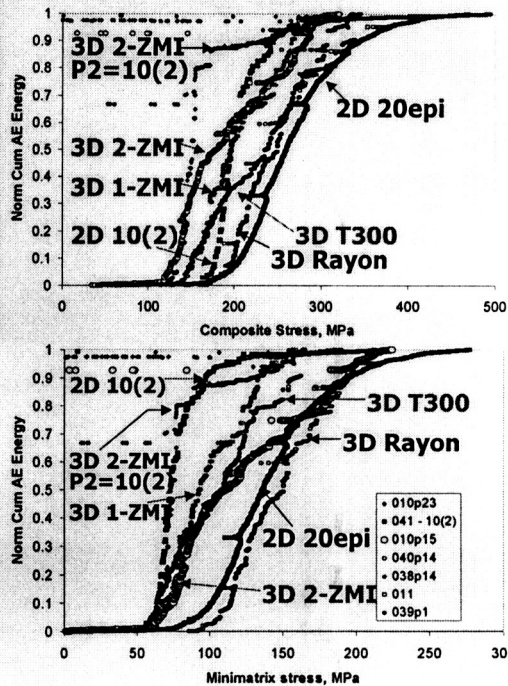
Specimen	f_{fiber}	f_{mini}	E_{mini}
Double ZMI	0.17	0.28	374
Single ZMI	0.23	0.33	356
T300	0.22	0.35	357
Rayon	0.20	0.35	363
20epi 2D	0.19	0.37	370

Comparison Between 2D and 3D Architectures "When Things Happen" (Normalized AE Activity)

For tiny Z bundle (Rayon), 3D matrix cracking very similar to 2D matrix cracking

For large 0° bundle (P2), 3D matrix cracking very similar to 2D matrix cracking → Narrower and lower stress range

For larger Z bundle sizes, 3D matrix cracking occurs at lower matrix stresses than 2D



Conclusions

- Z-bundles act as flaws for first matrix cracks in 3D composites
 - The smaller the Z-bundle the higher the stress for matrix cracking. However, for tows greater in size than Rayon, detection of "loud" AE (large matrix cracks) occurs at about the same stress.
 - Good for thermal conductivity? A debit to interlaminar properties?
- Stress-induced matrix cracking in 3D orthogonal composites:
 - Is dependent on the local section of the architecture for composites oriented in the Y-direction...
 - UNI matrix cracking occurs over a narrow, lower stress-range
 - XPLY matrix cracking occurs over a wider, higher stress-range
 - Occurred over a relatively narrow stress-range when oriented in the X-direction compared to Y-direction oriented composites
 - However, this could be due to 10(2) orientation
- Stress-dependence for matrix cracking in 3D orthogonal composites is very similar to 2D fabric when:
 - 0° tows are large, i.e., 10(2)
 - Z-bundle is very small for Y-direction orientation, e.g., Rayon

Implications

- **Property/architecture trade-offs will have to be considered in using these 3D architectures for elevated temperature applications:**
 - Lower stresses for cracking due to larger Z-bundle size will lower the stresses these materials will be able to withstand under oxidizing conditions at intermediate temperatures (600 to 1000°C)
 - Efforts to increase matrix cracking stress with smaller Z-bundle sizes will lower through-thickness strength and toughness
- **Approach to modeling matrix cracking in melt-infiltrated SiC/SiC 2D composites should be able to be extended to these 3D architectures**